

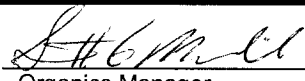
CT&E Environmental Services Inc.

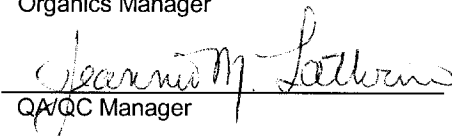
Standard Operating Procedure

Document Title: PCB Water Extraction
Method Reference: SW 3510C
Document File Name: 4314-03.DOC
Revision Number: 3.0
Effective Date: February 21, 2002

Document Control Number: 4314

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Approved by:  2-21-02
Organics Manager Date

Approved by:  2-21-02
QA/QC Manager Date

1.0 Scope and Applications

- 1.1 This method describes a procedure for isolating polychlorinated biphenyls (PCBs) from an aqueous sample.
- 1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of Method

- 2.1 A measured volume of sample, usually 0.8 liter, is extracted at a pH range of 5 to 9 with methylene chloride in a separatory funnel. The extract is concentrated and exchanged into hexane. Further cleanup is performed as required.

3.0 Interferences

- 3.1 Solvents, reagents, incorrectly cleaned glassware, and other extraction techniques may yield interference components in the final extract.
- 3.2 Care should be taken in cleaning any extraction vessel or equipment that the sample or sample extract may contact during the extraction process. Clean all glassware with Alconox[®] and hot water, rinse with DI water, acetone and methylene chloride.
- 3.3 Interferences co-extracted from samples vary from matrix to matrix and from sample to sample. Additional cleanup (acid cleanup, sulfur cleanup) may be required as needed or specified by project.
- 3.4 Contamination or carry-over may occur by extraction of high level samples sequentially with clean or low level samples. Proper record keeping allows for review of how and in what order samples were extracted if a high level sample is discovered during analysis. This makes it possible to determine which samples may have carry over and need re-extraction.

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4.0 Apparatus

- 4.1 Turbo Vap[®] tubes
- 4.2 Separatory funnels: 1 or 2 liter, Teflon[®] or glass with Teflon[®] stopcock.
- 4.3 Pipettes: 1.0 ml with 0.01 ml indicators
- 4.4 Pipettes: transfer
- 4.5 Glas-Col 3D[®] autoshaker, 10 place
- 4.6 Zymark TurboVap II[®]
- 4.7 Pyrex[®] glass wool, baked at 350°C for 3 hours.

5.0 Reagents

- 5.1 Reagent or pesticide grade chemicals are to be used in all extractions.
 - 5.1.1 Sodium sulfate, Na_2SO_4 , baked at 400°C for 4 hours.
 - 5.1.2 Hexane, C_6H_{12}
 - 5.1.3 Methylene chloride, CH_2Cl_2
 - 5.1.4 Water - all references to water refer to organic-free reagent water.
 - 5.1.5 Sodium Hydroxide solution (10N), NaOH, Dissolve 40g NaOH in water and dilute to 100ml.
 - 5.1.6 Sulfuric Acid solution (1:1 v/v), H_2SO_4 , SLOWLY add 50 ml of H_2SO_4 to 50 ml water.
- 5.2 All reagents and solvents are assigned tracking numbers and the numbers recorded in the solvent tracking log upon arrival in the lab. These numbers are then recorded in the extraction log with the associated samples.

6.0 Sample Extract Handling

- 6.1 Sample should be stored at 4°C ($\pm 2^\circ \text{C}$) until extraction. Water samples must be extracted within 7 days of sampling.
- 6.2 Sample extracts should be refrigerated.

7.0 Procedure

- 7.1 Clean all glassware thoroughly with Alconox[®] and hot water, rinse with tap water, DI water, acetone, and methylene chloride.
- 7.2 Using a 1 liter graduated cylinder, measure 0.8 liter of sample and transfer it to the separatory funnel. If high concentrations are expected, a smaller volume may be used and diluted with organic-free reagent water to 0.8 liter. Add 1.0 ml of appropriate surrogate to separatory funnel. Add 1.0 ml of PCB spike mix 1254 at 5

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µg/ml to all LCS, BSD, MS and MSD. Record starting volume, and surrogate tracking number in runlog. Record date, extraction type, analyst initials, client ID, sample number and initial volume on tag attached to separatory funnel.

- 7.3 Check pH with wide-range pH paper and record in extraction log. Adjust pH to a range of 5 to 9 with 10N NaOH or 1:1 H₂SO₄.
- 7.4 Add 60 ml methylene chloride to graduated cylinder and transfer to the separatory funnel.
- 7.5 Seal and shake for 2 minutes with periodic venting to release excess pressure.
 - 7.5.1 Seal the separatory funnel, manually shake once and vent into the hood. Load the separatory funnel on the 3D[®] autoshaker and clamp securely while checking for leaks.
 - 7.5.2 Set the 3D[®] autoshaker timer to 2 minutes and the shaking setting to between 50 and 70 (this range should be sufficient for vigorous shaking).
- 7.6 Allow the organic layer to separate from the water layer for a minimum of 10 minutes. If the emulsion interface between the layers is more than 1/3 the size of the solvent layer, mechanical techniques must be employed to reduce the interface. This may include stirring, centrifugation, or filtration of the emulsion through glass wool.
- 7.7 Drain the solvent (bottom layer) through a drying funnel containing anhydrous sodium sulfate with a glass wool stopper. Collect in a TurboVap[®] tube.
- 7.8 Repeat the extraction two more times with fresh solvent. Rinse powder funnel with methylene chloride after the third extraction. Combine the three solvent extracts in a TurboVap[®] tube. Transfer the tag to the TurboVap[®] tube.
- 7.9 Reduce and exchange the extract to hexane.
 - 7.9.1 Set TurboVap[®] temperature to 50°C and gas pressure between 8-15 psi.
 - 7.9.2 Place tube in TurboVap[®] and reduce to 5 ml, rinse down the walls of the tube with 50 ml of hexane. Reduce again and repeat the 50 ml rinse with hexane.
 - 7.9.3 Periodically use a transfer pipette to rinse down the inner walls of the tube with the extract to further concentrate compounds of interest into the final volume.

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- 7.11 Allow the solvent to reduce to 5 ml and transfer quantitatively to a 10 ml glass vial with a Teflon[®] screw cap. Record the final volume in the run log and on the tag attached to the sample. Reduce final volume to 1.0 ml when low-level analysis is required.

8.0 Quality Control

- 8.1 Prepare a method blank, LCS, BSD, MS, and MSD for each set of up to 20 samples. Refer to document control number 4106, § 10.0. Record Lab ID, extraction date, analyst initials, sample volumes, and any other characteristics on sample tag and in extraction log.
- 8.2 Add 1.0 ml of the appropriate surrogate mix to every sample and the Prep blank, LCS, BSD, MS, and MSD. Add 1.0 ml of the appropriate spike mix to the LCS, BSD, MS and MSD. Refer to document control number 4106, § 14.4, Table 2 for spike descriptions and concentrations. Record surrogate and spike concentrations, volumes, and tracking numbers in extraction log.

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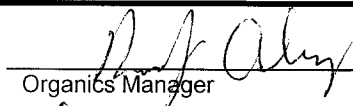
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Document Title: PCB Water Extraction
Method Reference: SW 3510C modified
Document File Name: 4327-01.DOC
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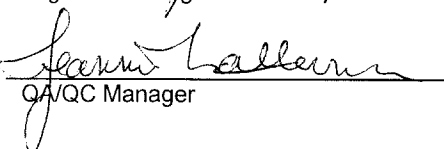
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Approved by:


Organics Manager

10-27-04
Date

Approved by:


QA/QC Manager

10-27-04
Date

1.0 Scope and Applications

- 1.1 This method describes a procedure for isolating polychlorinated biphenyls (PCBs) from an aqueous sample.
- 1.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

2.0 Summary of Method

- 2.1 A measured volume of sample, 2.0 liters, is extracted at a pH range of 5 to 9 with methylene chloride in a separatory funnel. The extract is concentrated and exchanged into hexane. Further cleanup is performed as required.

3.0 Interferences

- 3.1 Solvents, reagents, incorrectly cleaned glassware, and other extraction techniques may yield interference components in the final extract.
- 3.2 Care should be taken in cleaning any extraction vessel or equipment that the sample or sample extract may contact during the extraction process. Clean all glassware with Alconox[®] and hot water, rinse with DI water, acetone and methylene chloride.
- 3.3 Interferences co-extracted from samples vary from matrix to matrix and from sample to sample. Additional cleanup (acid cleanup, sulfur cleanup) may be required as needed or specified by project.
- 3.4 Contamination or carry-over may occur by extraction of high level samples sequentially with clean or low level samples. Proper record keeping allows for review of how and in what order samples were extracted if a high level sample is discovered during analysis. This makes it possible to determine which samples may have carry over and need re-extraction.

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4.0 Apparatus

- 4.1 Turbo Vap[®] tubes
- 4.2 Separatory funnels: 2 liter, Teflon[®] or glass with Teflon[®] stopcock.
- 4.3 Pipettes: 1.0 ml with 0.01 ml indicators
- 4.4 Pipettes: transfer
- 4.5 Glas-Col 3D[®] auto shaker, 10 place
- 4.6 Zymark TurboVap II[®]
- 4.7 Pyrex[®] glass wool, baked at 350°C for 3 hours.

5.0 Reagents

- 5.1 Reagent or pesticide grade chemicals are to be used in all extractions.
 - 5.1.1 Sodium sulfate, Na_2SO_4 , baked at 400°C for 4 hours.
 - 5.1.2 Hexane, C_6H_{12}
 - 5.1.3 Methylene chloride, CH_2Cl_2
 - 5.1.4 Water - all references to water refer to organic-free reagent water.
 - 5.1.5 Sodium Hydroxide solution (10N), NaOH, Dissolve 40g NaOH in water and dilute to 100ml.
 - 5.1.6 Sulfuric Acid solution (1:1 v/v), H_2SO_4 , SLOWLY add 50 ml of H_2SO_4 to 50 ml water.
- 5.2 All reagents and solvents are assigned tracking numbers and the numbers recorded in the solvent tracking log upon arrival in the lab. These numbers are then recorded in the extraction log with the associated samples.

6.0 Sample Extract Handling

- 6.1 Sample should be stored at 4°C ($\pm 2^\circ \text{C}$) until extraction. Water samples must be extracted within 7 days of sampling.
- 6.2 Sample extracts should be refrigerated.

7.0 Procedure

- 7.1 Clean all glassware thoroughly with Alconox[®] and hot water, rinse with tap water, DI water, acetone, and methylene chloride.
- 7.2 Using a 1 liter graduated cylinder, measure two 1 liter portions of sample and transfer it to a 2 liter separatory funnel. If high concentrations are expected, a smaller volume may be used and diluted with organic-free reagent water. Add 0.1

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ml of appropriate surrogate to separatory funnel. Add 0.1 ml of PCB spike mix 1254 at 5 µg/ml to all LCS, BSD, MS and MSD. Record starting volume and surrogate tracking number in run log. Record date, extraction type, analyst initials, client ID, sample number and initial volume on tag attached to separatory funnel.

- 7.3 Check pH with wide-range pH paper and record in extraction log. Adjust pH to a range of 5 to 9 with 10N NaOH or 1:1 H₂SO₄.
- 7.4 Add 60 ml methylene chloride to graduated cylinder and transfer to the separatory funnel.
- 7.5 Seal and shake for 2 minutes with periodic venting to release excess pressure.
 - 7.5.1 Seal the separatory funnel, manually shake once and vent into the hood. Load the separatory funnel on the 3D[®] auto shaker and clamp securely while checking for leaks.
 - 7.5.2 Set the 3D[®] auto shaker timer to 2 minutes and the shaking setting to between 50 and 70 (this range should be sufficient for vigorous shaking).
- 7.6 Allow the organic layer to separate from the water layer for a minimum of 10 minutes. If the emulsion interface between the layers is more than 1/3 the size of the solvent layer, mechanical techniques must be employed to reduce the interface. This may include stirring, centrifugation, or filtration of the emulsion through glass wool.
- 7.7 Drain the solvent (bottom layer) through a drying funnel containing anhydrous sodium sulfate with a glass wool stopper. Collect in a TurboVap[®] tube.
- 7.8 Repeat the extraction two more times with fresh solvent. Rinse powder funnel with methylene chloride after the third extraction. Combine the three solvent extracts in a TurboVap[®] tube. Transfer the tag to the TurboVap[®] tube.
- 7.9 Reduce and exchange the extract to hexane.
 - 7.9.1 Set TurboVap[®] temperature to 50°C and gas pressure between 8-15 psi.
 - 7.9.2 Place tube in TurboVap[®] and reduce to 5 ml, rinse down the walls of the tube with 50 ml of hexane. Reduce again and repeat the 50 ml rinse with hexane.

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7.9.3 Periodically use a transfer pipette to rinse down the inner walls of the tube with the extract to further concentrate compounds of interest into the final volume.

7.11 Allow the solvent to reduce to 1 ml and transfer quantitatively to a 10 ml glass vial with a Teflon[®] screw cap. Record the final volume in the run log and on the tag attached to the sample.

8.0 Quality Control

8.1 Prepare a method blank, LCS, BSD, MS, and MSD for each set of up to 20 samples. Refer to document control number 4106, § 10.0. Record Lab ID, extraction date, analyst initials, sample volumes, and any other characteristics on sample tag and in extraction log.

8.2 Add .01 ml of the appropriate surrogate mix to every sample and the Prep blank, LCS, BSD, MS, and MSD. Add 0.1 ml of the appropriate spike mix to the LCS, BSD, MS and MSD. Refer to document control number 4328, § 14.4, Table 3 for spike descriptions and concentrations. Record surrogate and spike concentrations, volumes, and tracking numbers in extraction log.

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Standard Operating Procedure

Document Title: PCB Analysis by Method 8082
Method Reference: SW846, Method 8082 (General Electric/Columbus Gas)
Document File Name: 4131-01.DOC
Revision Number: 1.0
Effective Date: October 10, 2003

Document Control Number: 4131

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Approved by: _____

Organics Manager

10-10-2003

Date

Approved by: _____

QA/QC Manager

10-10-2003

Date

1.0 Scope and Application

This procedure is used to determine the concentration of various polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices. Specifically it may be used to detect the following PCB Aroclors:

Table 1. PCB Aroclors

AROCLOR	LOQ (µg/L)	LOQ (µg/Kg)	Estimated Response Time in minutes			
			7*	14*	15*	Avg.
PCB-1016	0.065	33	2.679	2.350	3.123	2.7173
PCB-1221	0.065	33	1.680	1.566	2.118	5.364
PCB-1232	0.065	33	2.669	2.350	3.074	2.698
PCB-1242	0.065	33	2.407	2.350	3.061	2.606
PCB-1248	0.065	33	2.713	2.350	3.083	2.715
PCB-1254	0.065	33	5.199	5.128	5.917	5.415
PCB-1260	0.065	33	6.617	7.217	7.133	6.989

* GC ID numbers. These response times will vary and are only listed here for reference purposes.

SGS analyzes PCB-1262 and PCB-1268 for reference only.

2.0 Summary of Method

This method contains chromatographic conditions for the detection of PCBs. Prior to the use of this method, appropriate extraction and clean up techniques must be performed. Groundwater and other aqueous samples are extracted with methylene chloride using a separatory funnel (method SW3510C – refer to SOP 4314). Solid samples are extracted with acetone-hexane by soxhlet (method SW3541 – refer to SOP 4308). Oil samples are diluted with hexane (method SW3580A – refer to SOP 4321). A 2.0 µl sample is autoinjected into the gas chromatograph and the halogenated components are detected by an electron capture detector (ECD).

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3.0 Interferences

- 3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks.
- 3.2 Interferences co-extracted from the samples will vary considerable from sample to sample. While general clean up techniques are performed during the extraction process, unique samples may require additional clean up.
- 3.3 Clean all glassware prior to use with soap and water, rinse with DI water. Before use, the glassware is rinsed once with acetone and rinsed twice with methylene chloride.
- 3.4 Interferences by phthalate esters may pose a problem in the detection of PCBs. Minimizing contact with any type of plastic material will help to avoid this interference.
- 3.5 Contamination by carry over can occur whenever high-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross contamination.

4.0 Sample Handling

- 4.1 Upon receipt, all samples are refrigerated at 4°C (\pm 2°C) until extraction time.
- 4.2 Holding Times: Water samples must be extracted within seven (7) days of sampling; soil, solid, and wipe samples within fourteen (14) days, and oil samples have no holding time. All others must be characterized before a holding time can be determined. The extracts must be analyzed within forty (40) days following extraction. Sample extracts should be stored at 4°C (\pm 2°C) until time of analysis.
- 4.3 Post Analysis: refer to § 9.3

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5.0 Apparatus

5.1 Instrument:

Varian Model 3400 gas chromatograph with dual electron capture detector, single tower 8100 autoinjector to EZChrom V6.5 software.

5.2 Columns:

Primary: 15M, JW DB-1701 0.53 mm, ID 1.0 um film megabore capillary or equivalent.

Secondary: 15M, JW DB-608 0.53 mm, ID 0.83 um film megabore capillary or equivalent.

5.3 Carrier Gas: Nitrogen, 20 ml/min total flow

5.4 Glassware: The glassware used is dependent on the extraction method used. The glassware for a specific method is stated at the beginning of the extraction procedure.

5.5 2 ml Snap-Loc glass autosampler vials.

6.0 Reagents

Refer to Table 2 (§ 7.1.3) for more details on ICALs, standards, surrogates and spikes. All standards, etc. expire six (6) months from the date they are opened or prepared, but not to exceed the manufacture's expiration date. They must be stored at 4°C (\pm 2°C).

6.1 Stock Standards: Purchased certified solutions of PCBs and Surrogates in ampoules, refer to manufacture's expiration date. Store stock standards at 4°C (\pm 2°C).

6.2 Intermediate Standards: Intermediate standards (refer to Table 2, §7.1.3) are prepared by diluting stock standards. Preparation of intermediate standards should be done volumetrically using clean glassware. Store intermediate standards at 4°C (\pm 2°C) and check for evaporation before using. Opened standards are good for six (6) months.

6.3 Calibration Standards: A minimum of five (5) standards for each analyte and surrogate should be prepared from intermediate standards (refer to Table 2,

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§7.1.3). Different sources for the calibration and spike standards are used (this is accomplished by using different lot numbers).

- 6.4 Mineral oil – shelf life of two years.
- 6.5 Hexane (JT Baker) – shelf life of two years.
- 6.6 Acetone (JT Baker) – shelf life of two years.
- 6.7 Gauze Pads, 4 inches x 4 inches, sterile, cellulose base (Certified Safety Manufacturing Company)
- 6.8 Sulfuric Acid (JT Baker) – shelf life (Manufacturers Expiration)
- 6.9 Tetrabutylammonium hydrogen sulfite (JT Baker) – shelf life (Manufacturers Expiration)
- 6.10 Sodium Sulfate (JT Baker)
- 6.11 Tetrabutylammonium (TBA) sulfite reagent
 - 6.11.1 Tetrabutylammonium hydrogen sulfate, $[\text{CH}_3(\text{CH}_2)_3]\text{NHSO}_4$.
 - 6.11.2 Sodium sulfite, Na_2SO_3 .
 - 6.11.3 Prepare reagent by dissolving 13.6 g tetrabutylammonium hydrogen sulfate, and 100 g sodium sulfite in 400 ml of organic free water, in a 800 ml separatory funnel. Shake vigorously for 1 min. Observe the reagent to make sure all the solids are dissolved. To remove impurities, add 400 ml of hexane, recap, and shake for 1 min. Drain off the water layer (lower), and store at room temperature, in an amber bottle with a PTFE-lined screw cap. Discard the leftover hexane.

7.0 Calibration

7.1 Initial Calibration

- 7.1.1 Analyze two (2) hexane blanks which will pre-condition the GC column and provide a check to determine possible contamination. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.
- 7.1.2 For each analyte prepare calibration standard at a minimum of five (5) concentration levels by volumetric dilutions. The lowest standard should be at, or below the limit of quantitation (LOQ). The others should define the

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working range of the detector and give a linear calibration. Each column is calibrated using standards at concentrations listed in Table 2 (§ 7.1.3) below.

- 7.1.3 Analyze each calibration standard using the same technique as for samples. A minimum of three congeners for each Aroclor pattern is measured and an average response factor (ARF) is calculated for each congener by the data processing software after calibration is complete. Please refer to §11.5 for the ARF calculation. The % RSD must be less than 20% for the initial calibration to be acceptable. If this criteria is not met, recalibration of each failing Aroclor is required.

Table 2. PCB Standard Concentrations (in µg/ml)

AROCLOR	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6
1016	0.04	0.08	0.2	0.8	1.0	2.0
1221	0.04	0.08	0.2	0.8	1.0	2.0
1232	0.04	0.08	0.2	0.8	1.0	2.0
1242	0.04	0.08	0.2	0.8	1.0	2.0
1248	0.04	0.08	0.2	0.8	1.0	2.0
1254	0.04	0.08	0.2	0.8	1.0	2.0
1260	0.04	0.08	0.2	0.8	1.0	2.0

- 7.1.4 Retention Time Windows: Establish retention time windows for each analyte. Make three (3) injections of the CCAL over a 72-hour period and note the retention time of each measured congener. Calculate the standard deviation. The retention time of each congener for positive identification must fall within the ± 3 (STD) of a daily CCALRT.

- 7.1.5 The averaging of the selected congeners quantitates the total response of each PCB or Aroclor.

7.2 Daily:

- 7.2.1 Analyze the two (2) hexane blanks which will pre-condition the GC column and provide a check to determine possible contamination. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

- 7.2.2 Following the two (2) hexane blanks, analyze the mid-level (0.8 µg/ml) calibration standards for the following Aroclors: PCB 1242/1268, 1016/1260, 1232/1262, 1254/SURR, 1221, 1248. Calculate the %D for

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PCB 1254 and 1016/1260 against the initial calibration ARF. All CCALs in the beginning of the day must be checked for passing. If they do not pass, reanalyze the CCALs or recalibrate as appropriate; otherwise, these Aroclors may not be reported as "hits" for that day.

Note: PCB-1262 and PCB-1268 are run for reference only.

- 7.2.3 If the hexane blanks do not indicate any contamination problems, and the instrument passes calibration for PCB 1254, PCB 1016, PCB 1260, establish the daily run sequence.

8.0 Extraction

8.1 Liquid samples: Follow procedure in method SW3510C – SOP 4314.

8.2 Solid samples: Follow procedure in method SW3541 – SOP 4308.

8.3 Oil samples: Follow procedure in method SW3580A – SOP 4321.

8.4 Post Extraction Sample Preparation:

8.4.1 Sulfuric Acid Cleanup:

8.4.1.1 Using a Repipetter, transfer approximately 1 ml of concentrated sulfuric acid/water solution to the extracted sample.

Caution: Make sure there is no exothermic reaction, or evolution of gas prior to proceeding

8.4.1.2 Cap the vial tightly and shake vigorously for approximately 1 minute.

8.4.1.3 Allow the phases to separate (spin in centrifuge when needed). Examine the top (hexane) layer; it should not be highly colored or should it have a visible emulsion or cloudiness.

8.4.1.4 If clean phase separation is achieved, proceed to §8.4.1.6.

8.4.1.5 If the hexane layer is not clean, using a transfer pipette, transfer the hexane layer to a clean 10 ml glass PTFE lined screw-cap vial. Add another 1 ml portion of concentrated sulfuric acid/water solution and perform another acid cleanup, beginning at §8.4.1.1.

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8.4.1.6 Transfer the cleaned hexane to a 10 ml vial containing approximately 1 ml of organic free water.

8.4.1.7 Cap the vial tightly and shake vigorously for approximately 1 minute.

8.4.1.8 Allow the phases to separate.

8.4.1.9 Transfer the cleaned hexane to a 10 ml vial containing approximately 0.5 g of baked sodium sulfate.

8.4.1.10 Cap and shake. Proceed to §8.4.2.

8.4.2 Sulfur cleanup using Tetrabutylammonium hydrogen sulfite:

8.4.2.1 After performing any matrix specific dilutions on the acid cleaned sample; in a clean 10 ml vial mix 1 ml TBA solution with 2 ml of 2-Propanol. Cap and shake vial to mix.

8.4.2.2 To this sulfur cleaning mixture, add approximately 3 ml of acid cleaned sample.

8.4.2.3 Cap the vial tightly and shake vigorously for approximately 1 minute.

8.4.2.4 If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 1.00 g portions until a solid residue remains after repeated shaking.

8.4.2.5 Fill the vial, leaving some headspace, with organic free water and shake for approximately 1 minute.

8.4.2.6 Analyze the hexane layer accordingly.

9.0 Analysis

9.1 Establish the Daily Run Sequence

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The following is the required sequencing to be used by the analyst when loading the GC autosampler. Since the analysis is single injection with dual column dual detector, the analytical sequence is the same for both columns.

- 9.1.1 After calibrations have run (see §7.2) and all acceptance criteria has been met, an instrument blank should be analyzed to determine if any carry-over from the calibration standards exists. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

Note: MS, MSD and LCS should be run before an instrument blank in order to prevent contamination of other samples. The laboratory QC samples, LCS and MB, must be analyzed prior to or with the associated field samples in order to verify the sample preparation was acceptable.

- 9.1.2 Each sample analysis must be bracketed with a CCAL after every 12 hours or after every ten sample injections, whichever is more frequent. Therefore, after the instrument blank, up to ten (10) samples can be analyzed followed by a check standard and surrogate CCAL. This procedure should be followed until all samples are loaded. The CCALs should alternate between a PCB 1254 standard and a PCB 1016 and PCB 1260 standard mix. Bracketing CCALs must also meet 15%D as in § 7.2.2 above to be acceptable. If this criterion is not met the samples analyzed before and after the failing CCAL must be reanalyzed. The only exception would be if the samples are non-detect and the instrument is demonstrating increased sensitivity (CCALs failing high).
- 9.1.3 Complete the handwritten run log for the run sequence.
- 9.1.4 Type the run sequence into the data system.
- 9.1.5 Examine the computer sequence and verify this batch sequence with the run log and the sample tray.
- 9.1.6 Start the autosampler.
- 9.1.7 If additional samples are added after the batch sequence has been established, the analyst must verify the run order of the sample tray with the handwritten run log upon completion of the sequence. Regardless, the analytical sequence must end with a CCAL.

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9.1.8 Before emptying the sample wheel, check data runs with run log and sample placement in wheel. This is to ensure that no errors in data acquisition occurred during overnight sampling sequences.

9.2 Data Analysis

PCBs are multi-component compounds (congener mix and pattern) and are closely compared to their respective CCAL standards. However, the analyst's experience and skill in identification is more heavily relied upon due to PCBs being routinely weathered and chromatographically interfered with.

9.2.1 Peaks (congeners) which fall within the retention time window, are compared to known standards for identification of PCBs. Qualitative and quantitative analyses are confirmed by co-analysis on the alternate column. Qualitative confirmation is achieved if chromatographic patterns from both columns match those of the CCALs. PCBs are confirmed quantitatively if the RPD between the two analyses is less than 40%. If the %RPD is <40, report the greater concentration of the two columns. If the %RPD is >40, and the analyst in their technical opinion believes the variance is due to positive chromatographic interference, the lesser of the concentration of the two columns is reported. If this cannot be demonstrated, the greater concentration is reported.

9.2.2 Cross-checking the chains of custody and close coordination with GC prep are necessary to ensure that all requirements and method specifications are met.

9.3 Post Analysis

Retain sample extract vials for 40 days from date of extraction. Separate extracts for disposal by >35 ppm and <35 ppm. A list of samples with PCBs >35 ppm is given to GC prep and the sample custodian so that they may segregate them. These samples, and extracts, must be disposed of as PCB contaminated. All non-PCB samples and extracts are disposed of as non-chlorinated waste. Refer also to §13.0.

10.0 Quality Control

10.1 **Initial Calibration** (See § 7.1)

10.2 **Qualitative Verification Standards:** A qualitative verification (response, retention time and pattern) of all Aroclors must be verified each working day by analyzing

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standards of Aroclors 1221, 1232, 1248 and 1242 at 0.8 ppm. These standards are analyzed at the beginning of every instrument sequence.

- 10.3 **Continuing Calibration Verification Standard (CCAL):** The working calibration curve must be verified by analyzing CCALs. The CCALs are Aroclors 1016/1260 and 1254 at 0.8 ppm. These standards are analyzed at the beginning of every analytical sequence and after every 10 injections alternating between Aroclors 1016/1260 and Aroclor 1254.

The % difference between the response factor of the initial CCALs and the average response factor from its corresponding initial calibration must be $\leq 15\%$ in order for sample analysis to proceed. When this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration again and proceeding with sample analysis. If routine maintenance does not return the instrument performance to meeting the QC requirements based on the last initial calibration, then a new initial calibration for the failing PCBs must be performed.

The % difference between the response factor of a bracketing and closing CCAL and the average response factor from its corresponding initial calibration must be $\leq 15\%$ to be acceptable. If this criterion is not met the samples analyzed before and after the failing CCAL must be reanalyzed. The only exception would be if the samples are non-detect and the instrument is demonstrating increased sensitivity (CCALs failing high).

- 10.4 **Surrogated Hexane blank:** Run a Hexane blank containing surrogate at 0.08 ppm after every CCAL. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.
- 10.5 **Surrogates:** The TCMX and DCBP surrogate standard is added to each sample, standard, matrix spike, matrix spike duplicate, method blank and laboratory control sample to measure the effectiveness and accuracy of the extraction procedure. The acceptance range for surrogate accuracy has been established as 40%-120% (unless other limits are required by the client or program). Only one surrogate is required to be acceptable. If both surrogates fall outside the acceptance window, re-prep and reanalysis is required.
- 10.6 **Method Blank:** At least one blank must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Values that exceed the LOQ indicate a contamination problem and require associated samples with a positive response be re-prepped and reanalyzed. The only exception would be those samples whose PCB response was 10X the concentration found in the method blank.

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- 10.7 **Laboratory Control Sample (LCS):** At least one LCS must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Recoveries of the LCS and LCS duplicate must be within 40%-120%, or within the limits established by projects, programs, and/or clients. If the LCS is outside acceptance criteria, all associated samples must be re-prepped and reanalyzed.

It is SGS's practice to spike the LCS with Aroclor 1254 as it is the most common Aroclor we detect. Other Aroclors, such as 1242, 1248, or 1260, may be used based on client's request. The LCS and LCS duplicate are correspondingly spiked with:

- 1.0 ml of 5.0 ppm spike solution for soils
- 1.0 ml of 5.0 ppm spike for oils and wipes
- 1.0 ml of 5.0 ppm spike solution for medium-level waters
- 0.2 ml of 5.0 ppm spike solution for low-level waters

- 10.8 **Matrix Spike Samples (MS/MSD):** At least one matrix spike and matrix spike duplicate must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Recoveries of the MS/MSD must be within 40%-130%, or within the limits established by projects, programs, and/or clients. Relative percent difference between the MS and MSD duplicate must be less than or equal to 40% to be acceptable. If the MS/MSD criterion is not met and the LCS recovery (§10.6) is acceptable, assume matrix interference. The samples do not require reanalysis. If the LCS is outside acceptance criteria, all associated samples must be re-prepped and reanalyzed.

- 10.9 **Data Reporting -** Prior to reporting to the client, data is reviewed by a peer in the Department for approval. The data is then automatically transferred to the Laboratory Information System (LIMS) for reporting.

- 10.10 If the instrument concentration of any compound detected in a sample exceeds the highest-level of the initial calibration, the sample extract must be diluted in order to achieve the concentration within the calibration range of the instrument.

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11.0 Calculations

- 11.1 The recoveries for LCSs and surrogates** are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{V_o}{V_e} \times 100 = \% \text{ recovery}$$

Where: V_o = the observed value
 V_e = the expected value

- 11.2 The calculation for MS/MSD spike recoveries** requires the subtraction of the sample contribution from the response of the spiked sample; the division of this result by the expected value of spike, and the multiplication by 100 yield a percent recovery.

$$\frac{V_o - S_c}{V_e} \times 100 = \% \text{ recovery}$$

Where: V_o = the observed value of the spiked sample
 V_e = the expected value
 S_c = the observed value of the sample

- 11.3 The relative percent difference** between MS/MSD samples, or between the two dissimilar columns, is calculated as the absolute difference between the MS and the MSD, or column A and column B, divided by the average of the sample and the duplicate, all multiplied by 100.

$$\frac{S_c - D_c}{[(S_c + D_c) \div 2]} \times 100 = \text{Relative Percent Difference (RPD)}$$

Where: D_c = observed duplicate sample concentration (MSD or result from Column B)
 S_c = the observed sample concentration (MS or result from Column A)

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- 11.4 Final Sample Concentration** is determined from the instrument concentration print out. The instrument calculation of final concentration is performed automatically by the following equation:

$$\frac{\text{Area} \times V_f \times DF}{\text{ARF} \times V_i} = \text{Final sample concentration in } \mu\text{g/L}$$

Where:

Area	= observed area count from peak integration
V _f	= final extract volume in ml
ARF	= average response factor generated by the initial calibration
V _i	= initial sample volume (in ml if aqueous or in grams if solid)
DF	= dilution factor

If a solid sample final concentration is to be reported against its percent solid content, the calculation is performed automatically by the LIMS according to the following equation:

$$\frac{F_c \times 100}{\%S} = \text{final sample concentration in } \mu\text{g/kg}$$

Where:

F _c	= final sample concentration in $\mu\text{g/Kg}$ from instrument as calculated above
%S	= percent solid content of sample

- 11.5 Average Response Factor (ARF)** is determined by the data processing software after calibration is complete. The instrument calculation of the ARF is performed automatically by the following equation:

$$\text{Response Factor (RF)} = \frac{\text{area count of PCB peak}}{\text{known concentration of PCB}}$$

$$\text{Average Response Factor (ARF)} = \frac{\text{sum of each response factor}}{\text{total number of concentration levels used}}$$

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11.6 Percent Relative Standard Deviation (%RSD):

$$\%RSD = \frac{SD}{RF_x} \times 100\%$$

Where: RSD = Relative standard deviation
RF_x = mean of 5 initial calibration factors for a compound
SD = standard deviation of the 5 initial calibration factors for a compound

12.0 Safety

Always wear gloves, safety glasses, and lab coats when performing this procedure. Be aware of the dangers involved with the solvents and reagents that you are using. The Material Safety and Data Sheets (MSDS) of all common lab reagents are available in the Quality Control Office.

13.0 Disposal of Samples, Standards, etc.

13.1 Segregate samples and standards, archive and submit to hazardous waste officer in accordance with policy outlined in quality control document.

13.2 **All samples and standards that are PCB positive must be segregated as PCB waste. Refer to §9.3**

14.0 Routine Maintenance

14.1 When gas pressure is about 500 psi, have a tank ready for replacement. Check for any leakage, particularly on joints, after installing a new gas tank.

14.2 Change septa daily or at analyst's discretion.

14.3 If signals are getting high and/or recoveries are inconsistent, do a thermal cleaning by setting oven temperature to 300°F and detectors to 320°F for 1-2 hours. If this step does not help, try clipping the columns. Clean-up by syonization may also be necessary. If none of these steps work, consider changing the columns. Other troubleshooting steps are referred to in the machine manual. Check for any possible leakage, as well as the total, column and split flows of gas, whenever the column is disconnected. For high baselines, check for contaminated gases.

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14.4 All maintenance procedures done on the instrument are to be recorded in the instrument's maintenance logbook.

Table 3. PCB ICAL, Surrogates and Spikes

Type	Description	Initial conc. µg/ml	Initial volume	Final conc. µg/ml	Final Volume	Solvent
ICAL All Aroclors except 1254	Intermediate ICAL at 50 µg/ml	1000 (of stock)	0.5 ml (of stock)	50	10 ml	Hexane
	ICAL mix at 2.0 µg/ml	50	2.0 ml	2.0	50 ml	Hexane
	ICAL mix at 1.0 µg/ml	50	1.0 ml	1.0	50 ml	Hexane
	ICAL mix at 0.8 µg/ml	50	0.8 ml	0.8	50 ml	Hexane
	ICAL mix at 0.2 µg/ml	50	0.2 ml	0.2	50 ml	Hexane
	ICAL mix at 0.08 µg/ml	50	0.08 ml	0.08	50 ml	Hexane
	ICAL mix at 0.04 µg/ml	50	0.04 ml	0.04	50 ml	Hexane
1254/Surrogate ICAL	Intermediate ICAL at 50/5 µg/ml	Aroclor 1254: 1000 (of stock)	Aroclor 1254: 0.5 ml (of stock)	Aroclor 1254: 50.0	10 ml	Hexane
		Surrogate: 200 (of stock)	Surrogate: 0.25 ml (of stock)	Surrogate: 5.0		
	ICAL mix at 2.0/0.2 µg/ml	Aroclor 1254: 50	Aroclor 1254: 2.0 ml	Aroclor 1254: 2.0	50 ml	Hexane
		Surrogate: 5	Surrogate: 2.0 ml	Surrogate: 0.2		
	ICAL mix at 1.0/0.1 µg/ml	Aroclor 1254: 50	Aroclor 1254: 1.0 ml	Aroclor 1254: 1.0	50 ml	Hexane
		Surrogate: 5	Surrogate: 1.0 ml	Surrogate: 0.1		
	ICAL mix at 0.8/0.08 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.8 ml	Aroclor 1254: 0.8	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.8 ml	Surrogate: 0.08		
	ICAL mix at 0.2/0.02 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.2 ml	Aroclor 1254: 0.2	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.2 ml	Surrogate: 0.02		
	ICAL mix at 0.08/ 0.008 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.08 ml	Aroclor 1254: 0.08	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.08 ml	Surrogate: 0.008		
	Ical mix at 0.04/ 0.004 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.04 ml	Aroclor 1254: 0.04	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.04 ml	Surrogate: 0.004		

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Table 3. PCB ICAL, Surrogates and Spikes

Type	Description	Initial conc. µg/ml	Initial volume	Final conc. µg/ml	Final Volume	Solvent
CCALs	CCAL mix at 0.8 µg/ml	1000 (of stock)	0.2 ml (of stock)	0.8	250 ml	Hexane
Surrogate for soil/water	PC Soil Water Surrogate	200 (of stock)	2.5 ml (of stock)	1.0	500 ml	Acetone
Surrogate for oils/wipes	PCB oil/wipe surrogate	200 (of stock)	2.0 ml (of stock)	0.1	4 L	Hexane
Spike	PCB Spike 1254 at 5 µg/ml	1000 (of stock)	2.5 ml (of stock)	5.0	500 ml	Acetone

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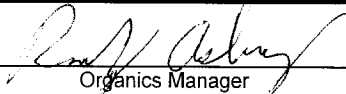
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Approved by:


Organics Manager

10-27-04

Date

Approved by:


QA/QC Manager

10-27-04

Date

1.0 Scope and Application

This procedure is used to determine the concentration of various polychlorinated biphenyls (PCBs) in groundwater and liquid sample matrices. Specifically it may be used to detect the following PCB Aroclors:

AROCLOR	LOQ (µg/L)	Estimated Response Time in Minutes Low Level Modified GC
PCB-1016	0.014	3.123
PCB-1221	0.014	2.118
PCB-1232	0.014	3.074
PCB-1242	0.014	3.061
PCB-1248	0.014	3.083
PCB-1254	0.014	5.917
PCB-1260	0.014	7.133

These response times will vary and are only listed here for reference purposes.

SGS analyzes PCB-1262 and PCB-1268 for reference only.

2.0 Summary of Method

This method contains chromatographic conditions for the detection of PCBs. Prior to the use of this method, appropriate extraction and clean up techniques must be performed. Groundwater and other aqueous samples are extracted with methylene chloride using a separatory funnel (method SW3510C – refer to SOP 4327). A 4.0 µl sample is auto injected into the gas chromatograph and the halogenated components are detected by an electron capture detector (ECD).

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3.0 Interferences

- 3.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks.
- 3.2 Interferences co-extracted from the samples will vary considerable from sample to sample. While general clean up techniques are performed during the extraction process, unique samples may require additional clean up.
- 3.3 Clean all glassware prior to use with soap and water, rinse with DI water. Before use, the glassware is rinsed once with acetone and rinsed twice with methylene chloride.
- 3.4 Interferences by phthalate esters may pose a problem in the detection of PCBs. Minimizing contact with any type of plastic material will help to avoid this interference.
- 3.5 Contamination by carry over can occur whenever high-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross contamination.

4.0 Sample Handling

- 4.1 Upon receipt, all samples are refrigerated at 4°C (\pm 2°C) until extraction time.
- 4.2 Holding Times: Water samples must be extracted within seven (7) days of sampling. The extracts must be analyzed within forty (40) days following extraction. Sample extracts should be stored at 4°C (\pm 2°C) until time of analysis.
- 4.3 Post Analysis: refer to § 9.3

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5.0 Apparatus

5.1 Instrument:

Varian Model 3400 gas chromatograph with dual electron capture detector, single tower 8100 auto injector to EZChrom V6.5 software.

5.2 Columns:

Primary: 15M, JW DB-1701 0.53 mm, ID 1.0 um film megabore capillary or equivalent.

Secondary: 15M, JW DB-608 0.53 mm, ID 0.83 um film megabore capillary or equivalent.

5.3 Carrier Gas: Nitrogen, 20 ml/min total flow

5.4 Glassware: The glassware used is dependent on the extraction method used. The glassware for a specific method is stated at the beginning of the extraction procedure.

5.5 2 ml Snap-Loc glass autosampler vials.

6.0 Reagents

Refer to Table 2 (§ 7.1.3) for more details on ICALs, standards, surrogates and spikes. All standards, etc. expire six (6) months from the date they are opened or prepared, but not to exceed the manufacture's expiration date. They must be stored at 4°C (± 2°C).

6.1 Stock Standards: Purchased certified solutions of PCBs and Surrogates in ampoules, refer to manufacture's expiration date. Store stock standards at 4°C (± 2°C).

6.2 Intermediate Standards: Intermediate standards (refer to Table 2, §7.1.3) are prepared by diluting stock standards. Preparation of intermediate standards should be done volumetrically using clean glassware. Store intermediate standards at 4°C (± 2°C) and check for evaporation before using. Opened standards are good for six (6) months.

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- 6.3 Calibration Standards: A minimum of five (5) standards for each analyte and surrogate should be prepared from intermediate standards (refer to Table 2, §7.1.3). Different sources for the calibration and spike standards are used (this is accomplished by using different lot numbers).
- 6.4 Hexane (JT Baker) – shelf life of two years.
- 6.5 Acetone (JT Baker) – shelf life of two years.
- 6.6 Sulfuric Acid (JT Baker) – shelf life (Manufacturers Expiration)
- 6.7 Tetrabutylammonium hydrogen sulfite (JT Baker) – shelf life (Manufacturers Expiration)
- 6.8 Sodium Sulfate (JT Baker)
- 6.9 Tetrabutylammonium (TBA) sulfite reagent
 - 6.9.1 Tetrabutylammonium hydrogen sulfate, $[\text{CH}_3(\text{CH}_2)_3]\text{NH}\text{SO}_4$.
 - 6.9.2 Sodium sulfite, Na_2SO_3 .
 - 6.9.3 Prepare reagent by dissolving 13.6 g tetrabutylammonium hydrogen sulfate, and 100 g sodium sulfite in 400 ml of organic free water, in an 800 ml separatory funnel. Shake vigorously for 1 min. Observe the reagent to make sure all the solids are dissolved. To remove impurities, add 400 ml of hexane, recap, and shake for 1 min. Drain off the water layer (lower), and store at room temperature, in an amber bottle with a PTFE-lined screw cap. Discard the leftover hexane.

7.0 Calibration

7.1 Initial Calibration

- 7.1.1 Analyze two (2) hexane blanks which will pre-condition the GC column and provide a check to determine possible contamination. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.
- 7.1.2 For each analyte prepare calibration standard at a minimum of five (5) concentration levels by volumetric dilutions. The lowest standard should be at, or below the limit of quantitation (LOQ). The others should define the working range of the detector and give a linear calibration. Each column is

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calibrated using standards at concentrations listed in Table 2 (§ 7.1.3) below.

- 7.1.3 Analyze each calibration standard using the same technique as for samples. A minimum of three congeners for each Aroclor pattern is measured and an average response factor (ARF) is calculated for each congener by the data processing software after calibration is complete. Please refer to §11.5 for the ARF calculation. The % RSD must be less than 20% for the initial calibration to be acceptable. If this criterion is not met, recalibration of each failing Aroclor is required.

Table 2. PCB Standard Concentrations (in µg/ml)

AROCLOR	LEVEL 1	LEVEL 2	LEVEL 3	LEVEL 4	LEVEL 5	LEVEL 6	LEVEL 7
1016	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1221	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1232	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1242	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1248	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1254	0.01	0.02	0.04	0.08	0.2	0.8	1.0
1260	0.01	0.02	0.04	0.08	0.2	0.8	1.0

- 7.1.4 Retention Time Windows: Establish retention time windows for each analyte. Make three (3) injections of the CCAL over a 72-hour period and note the retention time of each measured congener. Calculate the standard deviation. The retention time of each congener for positive identification must fall within the ± 3 (STD) of a daily CCALRT.

- 7.1.5 The averaging of the selected congeners quantitates the total response of each PCB or Aroclor.

7.2 Daily:

- 7.2.1 Analyze the two (2) hexane blanks which will pre-condition the GC column and provide a check to determine possible contamination. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

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- 7.2.2 Following the two (2) hexane blanks, analyze the mid-level (0.8 µg/ml) calibration standards for the following Aroclors: PCB 1242/1268, 1016/1260, 1232/1262, 1254/SURR, 1221, and 1248. Calculate the %D for PCB 1254 and 1016/1260 against the initial calibration ARF. All CCALs in the beginning of the day must be checked for passing. If they do not pass, reanalyze the CCALs or recalibrate as appropriate; otherwise, these Aroclors may not be reported as "hits" for that day.

Note: PCB-1262 and PCB-1268 are run for reference only.

- 7.2.3 If the hexane blanks do not indicate any contamination problems, and the instrument passes calibration for PCB 1254, PCB 1016, PCB 1260, establish the daily run sequence.

8.0 Extraction

- 8.1 Liquid samples: Follow procedure in method SW3510C – SOP 4327.

8.2 Post Extraction Sample Preparation:

8.2.1 Sulfuric Acid Cleanup:

- 8.2.1.1 Using a Repipetter, transfer approximately 1 ml of concentrated sulfuric acid/water solution to the extracted sample.

Caution: Make sure there is no exothermic reaction, or evolution of gas prior to proceeding

- 8.2.1.2 Cap the vial tightly and shake vigorously for approximately 1 minute.

- 8.2.1.3 Allow the phases to separate (spin in centrifuge when needed). Examine the top (hexane) layer; it should not be highly colored or should it have a visible emulsion or cloudiness.

- 8.2.1.4 If clean phase separation is achieved, proceed to §8.2.1.6.

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- 8.2.1.5 If the hexane layer is not clean, using a transfer pipette, transfer the hexane layer to a clean 10 ml glass PTFE lined screw-cap vial. Add another 1 ml portion of concentrated sulfuric acid/water solution and perform another acid cleanup, beginning at §8.2.1.1.
- 8.2.1.6 Transfer the cleaned hexane to a 10 ml vial containing approximately 1 ml of organic free water.
- 8.2.1.7 Cap the vial tightly and shake vigorously for approximately 1 minute.
- 8.2.1.8 Allow the phases to separate.
- 8.2.1.9 Transfer the cleaned hexane to a 10 ml vial containing approximately 0.5 g of baked sodium sulfate.
- 8.2.1.10 Cap and shake. Proceed to §8.2.2.
- 8.2.2 Sulfur cleanup using Tetrabutylammonium hydrogen sulfite:
 - 8.2.2.1 After performing any matrix specific dilutions on the acid cleaned sample; in a clean 10 ml vial mix 1 ml TBA solution with 2 ml of 2-Propanol. Cap and shake vial to mix.
 - 8.2.2.2 To this sulfur cleaning mixture, add approximately 3 ml of acid cleaned sample.
 - 8.2.2.3 Cap the vial tightly and shake vigorously for approximately 1 minute.
 - 8.2.2.4 If the sample is colorless or if the initial color is unchanged, and if clear crystals (precipitated sodium sulfite) are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 1.00 g portions until a solid residue remains after repeated shaking.
 - 8.2.2.5 Fill the vial, leaving some headspace, with organic free water and shake for approximately 1 minute.

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8.2.2.6 Analyze the hexane layer accordingly.

9.0 Analysis

9.1 Establish the Daily Run Sequence

The following is the required sequencing to be used by the analyst when loading the GC autosampler. Since the analysis is single injection with dual column dual detector, the analytical sequence is the same for both columns.

- 9.1.1 After calibrations have run (see §7.2) and all acceptance criteria has been met, an instrument blank should be analyzed to determine if any carry-over from the calibration standards exists. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

Note: MS, MSD and LCS should be run before an instrument blank in order to prevent contamination of other samples. The laboratory QC samples, LCS and MB, must be analyzed prior to or with the associated field samples in order to verify the sample preparation was acceptable.

- 9.1.2 Each sample analysis must be bracketed with a CCAL after every 12 hours or after every ten sample injections, whichever is more frequent. Therefore, after the instrument blank, up to ten (10) samples can be analyzed followed by a check standard and surrogate CCAL. This procedure should be followed until all samples are loaded. The CCALs should alternate between a PCB 1254 standard and a PCB 1016 and PCB 1260 standard mix. Bracketing CCALs must also meet 15%D as in § 7.2.2 above to be acceptable. If this criterion is not met the samples analyzed before and after the failing CCAL must be reanalyzed. The only exception would be if the samples are non-detect and the instrument is demonstrating increased sensitivity (CCALs failing high).
- 9.1.3 Complete the handwritten run log for the run sequence.
- 9.1.4 Type the run sequence into the data system.
- 9.1.5 Examine the computer sequence and verify this batch sequence with the run log and the sample tray.
- 9.1.6 Start the autosampler.

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9.1.7 If additional samples are added after the batch sequence has been established, the analyst must verify the run order of the sample tray with the handwritten run log upon completion of the sequence. Regardless, the analytical sequence must end with a CCAL.

9.1.8 Before emptying the sample wheel, check data runs with run log and sample placement in wheel. This is to ensure that no errors in data acquisition occurred during overnight sampling sequences.

9.2 Data Analysis

PCBs are multi-component compounds (congener mix and pattern) and are closely compared to their respective CCAL standards. However, the analyst's experience and skill in identification is more heavily relied upon due to PCBs being routinely weathered and chromatographically interfered with.

9.2.1 Peaks (congeners) which fall within the retention time window, are compared to known standards for identification of PCBs. Qualitative and quantitative analyses are confirmed by co-analysis on the alternate column. Qualitative confirmation is achieved if chromatographic patterns from both columns match those of the CCALs. PCBs are confirmed quantitatively if the RPD between the two analyses is less than 40%. If the %RPD is <40, report the greater concentration of the two columns. If the %RPD is >40, and the analyst in their technical opinion believes the variance is due to positive chromatographic interference, the lesser of the concentration of the two columns is reported. If this cannot be demonstrated, the greater concentration is reported.

9.2.2 Cross-checking the chains of custody and close coordination with GC prep are necessary to ensure that all requirements and method specifications are met.

9.3 Post Analysis

Retain sample extract vials for 40 days from date of extraction. Separate extracts for disposal by >35 ppm and <35 ppm. A list of samples with PCBs >35 ppm is given to GC prep and the sample custodian so that they may segregate them. These samples, and extracts, must be disposed of as PCB contaminated. All non-PCB samples and extracts are disposed of as non-chlorinated waste. Refer also to §13.0.

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10.0 Quality Control

10.1 Initial Calibration (See § 7.1)

10.2 **Qualitative Verification Standards:** A qualitative verification (response, retention time and pattern) of all Aroclors must be verified each working day by analyzing standards of Aroclors 1221, 1232, 1248 and 1242 at 0.8 ppm. These standards are analyzed at the beginning of every instrument sequence.

10.3 **Continuing Calibration Verification Standard (CCAL):** The working calibration curve must be verified by analyzing CCALs. The CCALs are Aroclors 1016/1260 and 1254 at 0.8 ppm. These standards are analyzed at the beginning of every analytical sequence and after every 10 injections alternating between Aroclors 1016/1260 and Aroclor 1254.

The % difference between the response factor of the initial CCALs and the average response factor from its corresponding initial calibration must be $\leq 15\%$ in order for sample analysis to proceed. When this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration again and proceeding with sample analysis. If routine maintenance does not return the instrument performance to meeting the QC requirements based on the last initial calibration, then a new initial calibration for the failing PCBs must be performed.

The % difference between the response factor of a bracketing and closing CCAL and the average response factor from its corresponding initial calibration must be $\leq 15\%$ to be acceptable. If this criterion is not met the samples analyzed before and after the failing CCAL must be reanalyzed. The only exception would be if the samples are non-detect and the instrument is demonstrating increased sensitivity (CCALs failing high).

10.4 **Surrogated Hexane blank:** Run a Hexane blank containing surrogate at 0.08 ppm after every CCAL. If any peaks appear in the appropriate retention time window for a PCB of interest, check for contamination and if present, reanalyze.

10.5 **Surrogates:** The TCMX and DCBP surrogate standard is added to each sample, standard, matrix spike, matrix spike duplicate, method blank and laboratory control sample to measure the effectiveness and accuracy of the extraction procedure. The acceptance range for surrogate accuracy has been established as 40%-120% (unless other limits are required by the client or program). Only one surrogate is required to be acceptable. If both surrogates fall outside the acceptance window, re-prep and reanalysis is required.

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10.6 **Method Blank:** At least one blank must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Values that exceed the LOQ indicate a contamination problem and require associated samples with a positive response be re-prepped and reanalyzed. The only exception would be those samples whose PCB response was 10X the concentration found in the method blank.

10.7 **Laboratory Control Sample (LCS):** At least one LCS must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Recoveries of the LCS and LCS duplicate must be within 40%-120%, or within the limits established by projects, programs, and/or clients. If the LCS is outside acceptance criteria, all associated samples must be re-prepped and reanalyzed.

It is SGS's practice to spike the LCS with Aroclor 1254 as it is the most common Aroclor we detect. Other Aroclors, such as 1242, 1248, or 1260, may be used based on client's request. The LCS and LCS duplicate are correspondingly spiked with:

- 0.1 ml of 5.0 ppm spike solution for low-level waters

10.8 **Matrix Spike Samples (MS/MSD):** At least one matrix spike and matrix spike duplicate must be taken through the entire process with each batch of samples (a batch is a maximum of 20 field samples of the same matrix prepared together). Recoveries of the MS/MSD must be within 40%-130%, or within the limits established by projects, programs, and/or clients. Relative percent difference between the MS and MSD duplicate must be less than or equal to 40% to be acceptable. If the MS/MSD criterion is not met and the LCS recovery (§10.6) is acceptable, assume matrix interference. The samples do not require reanalysis. If the LCS is outside acceptance criteria, all associated samples must be re-prepped and reanalyzed.

10.9 **Data Reporting -** Prior to reporting to the client, data is reviewed by a peer in the Department for approval. The data is then automatically transferred to the Laboratory Information System (LIMS) for reporting.

10.10 If the instrument concentration of any compound detected in a sample exceeds the highest-level of the initial calibration, the sample extract must be diluted in order to achieve the concentration within the calibration range of the instrument.

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11.0 Calculations

- 11.1 The recoveries for LCSs and surrogates** are calculated by dividing the observed value by the expected value. The result is multiplied by 100 to give a percent recovery.

$$\frac{V_o}{V_e} \times 100 = \% \text{ recovery}$$

Where: V_o = the observed value
 V_e = the expected value

- 11.2 The calculation for MS/MSD spike recoveries** requires the subtraction of the sample contribution from the response of the spiked sample; the division of this result by the expected value of spike, and the multiplication by 100 yield a percent recovery.

$$\frac{V_o - S_c}{V_e} \times 100 = \% \text{ recovery}$$

Where: V_o = the observed value of the spiked sample
 V_e = the expected value
 S_c = the observed value of the sample

- 11.3 The relative percent difference** between MS/MSD samples, or between the two dissimilar columns, is calculated as the absolute difference between the MS and the MSD, or column A and column B, divided by the average of the sample and the duplicate, all multiplied by 100.

$$\frac{S_c - D_c}{[(S_c + D_c) \div 2]} \times 100 = \text{Relative Percent Difference (RPD)}$$

Where: D_c = observed duplicate sample concentration (MSD or result from Column B)
 S_c = the observed sample concentration (MS or result from Column A)

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- 11.4 Final Sample Concentration** is determined from the instrument concentration print out. The instrument calculation of final concentration is performed automatically by the following equation:

$$\frac{\text{Area} \times V_f \times DF}{\text{ARF} \times V_i} = \text{Final sample concentration in } \mu\text{g/L}$$

Where:

Area	= observed area count from peak integration
V _f	= final extract volume in ml
ARF	= average response factor generated by the initial calibration
V _i	= initial sample volume (in ml if aqueous or in grams if solid)
DF	= dilution factor

- 11.5 Average Response Factor (ARF)** is determined by the data processing software after calibration is complete. The instrument calculation of the ARF is performed automatically by the following equation:

$$\text{Response Factor (RF)} = \frac{\text{area count of PCB peak}}{\text{known concentration of PCB}}$$

$$\text{Average Response Factor (ARF)} = \frac{\text{sum of each response factor}}{\text{total number of concentration levels used}}$$

- 11.6 Percent Relative Standard Deviation (%RSD):**

$$\%RSD = \frac{SD}{RF_x} \times 100\%$$

Where:

RSD	= Relative standard deviation
RF _x	= mean of 5 initial calibration factors for a compound
SD	= standard deviation of the 5 initial calibration factors for a compound

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12.0 Safety

Always wear gloves, safety glasses, and lab coats when performing this procedure. Be aware of the dangers involved with the solvents and reagents that you are using. The Material Safety and Data Sheets (MSDS) of all common lab reagents are available in the Quality Control Office.

13.0 Disposal of Samples, Standards, etc.

13.1 Segregate samples and standards, archive and submit to hazardous waste officer in accordance with policy outlined in quality control document.

13.2 All samples and standards that are PCB positive must be segregated as PCB waste. Refer to §9.3

14.0 Routine Maintenance

14.1 When gas pressure is about 500 psi, have a tank ready for replacement. Check for any leakage, particularly on joints, after installing a new gas tank.

14.2 Change septa daily or at analyst's discretion.

14.3 If signals are getting high and/or recoveries are inconsistent, do a thermal cleaning by setting oven temperature to 300°F and detectors to 320°F for 1-2 hours. If this step does not help, try clipping the columns. Clean-up by syonization may also be necessary. If none of these steps work, consider changing the columns. Other troubleshooting steps are referred to in the machine manual. Check for any possible leakage, as well as the total, column and split flows of gas, whenever the column is disconnected. For high baselines, check for contaminated gases.

14.4 All maintenance procedures done on the instrument are to be recorded in the instrument's maintenance logbook.

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Table 3. PCB ICAL, Surrogates and Spikes

Type	Description	Initial conc. µg/ml	Initial volume	Final conc. µg/ml	Final Volume	Solvent
ICAL All Aroclors except 1254	Intermediate ICAL at 50 µg/ml	1000 (of stock)	0.5 ml (of stock)	50	10 ml	Hexane
	ICAL mix at 1.0 µg/ml	50	1.0 ml	1.0	50 ml	Hexane
	ICAL mix at 0.8 µg/ml	50	0.8 ml	0.8	50 ml	Hexane
	ICAL mix at 0.2 µg/ml	50	0.2 ml	0.2	50 ml	Hexane
	ICAL mix at 0.08 µg/ml	50	0.08 ml	0.08	50 ml	Hexane
	ICAL mix at 0.04 µg/ml	50	0.04 ml	0.04	50 ml	Hexane
	ICAL mix at 0.02 µg/ml	50	0.02 ml	0.02	50 ml	Hexane
	ICAL mix at 0.01 µg/ml	50	0.01 ml	0.01	50 ml	Hexane
1254/Surrogate ICAL	Intermediate ICAL at 50/5 µg/ml	Aroclor 1254: 1000 (of stock)	Aroclor 1254: 0.5 ml (of stock)	Aroclor 1254: 50.0	10 ml	Hexane
		Surrogate: 200 (of stock)	Surrogate: 0.25 ml (of stock)	Surrogate: 5.0		
	ICAL mix at 1.0/0.1 µg/ml	Aroclor 1254: 50	Aroclor 1254: 1.0 ml	Aroclor 1254: 1.0	50 ml	Hexane
		Surrogate: 5	Surrogate: 1.0 ml	Surrogate: 0.1		
	ICAL mix at 0.8/0.08 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.8 ml	Aroclor 1254: 0.8	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.8 ml	Surrogate: 0.08		
	ICAL mix at 0.2/0.02 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.2 ml	Aroclor 1254: 0.2	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.2 ml	Surrogate: 0.02		
	ICAL mix at 0.08/ 0.008 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.08 ml	Aroclor 1254: 0.08	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.08 ml	Surrogate: 0.008		
	Ical mix at 0.04/ 0.004 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.04 ml	Aroclor 1254: 0.04	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.04 ml	Surrogate: 0.004		
	Ical mix at 0.02/ 0.002 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.02 ml	Aroclor 1254: 0.02	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.02 ml	Surrogate: 0.002		

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	Ical mix at 0.01/ 0.001 µg/ml	Aroclor 1254: 50	Aroclor 1254: 0.01 ml	Aroclor 1254: 0.01	50 ml	Hexane
		Surrogate: 5	Surrogate: 0.01 ml	Surrogate: 0.001		
CCALs	CCAL mix at 0.8 µg/ml	1000 (of stock)	0.2 ml (of stock)	0.8	250 ml	Hexane
Surrogate for water	PCB Water Surrogate	200 (of stock)	2.5 ml (of stock)	1.0	500 ml	Acetone
Spike	PCB Spike 1254 at 5 µg/ml	1000 (of stock)	2.5 ml (of stock)	5.0	500 ml	Acetone